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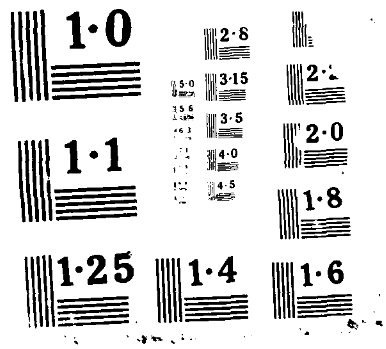
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High Temperature Creep Effects in Carbon Yarns and Composites

Prepared by

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5 December 1986

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Carbon yarns and composites were subjected to creep under tensile loading at 2000 to 3000°C. Changes in microstructure accompany changes in the apparent modulus of filaments and composites, as measured by a vibrating beam technique. Discussed are examples of decreased apparent modulus after high temperature creep and their implications for degree of fiber-matrix interfacial debonding.

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FOREWORD

The information in this report was included in an Extended Abstract for the 17th Biennial Conference on Carbon, held in Lexington, Kentucky, 16-21 June 1985.

We thank the Office of Naval Research for support in the preparation of this report and Dr. L. H. Peebles, Jr., for his encouragement and criticism.

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I. INTRODUCTION

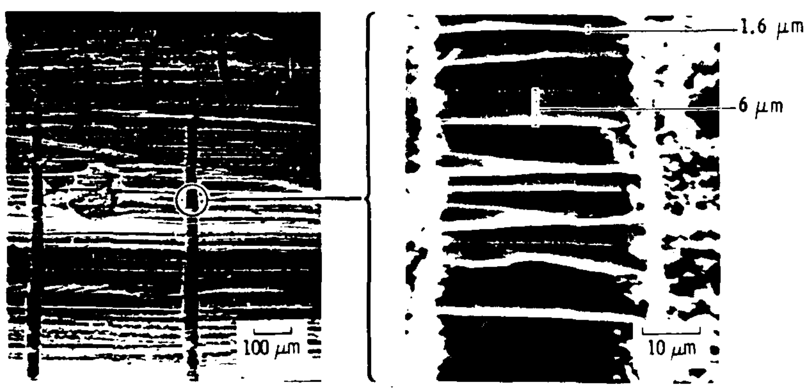
Carbon fibers and composites are increasingly being used in applications requiring strength and toughness at extremely high temperatures. But because high-modulus, highly aligned carbon fibers have large thermal expansion anisotropy, incorporating them into carbon-carbon composites requiring extreme processing temperatures induces large internal stresses in the material that can lead to damage and flaws. Stress relaxation can occur at high temperature by creep, the rate for which depends on temperature and stress.

The present investigation focuses on changes caused by creep under uniaxial tension in unidirectional composites of HM3000 PAN-based and P55 mesophase-pitch-based carbon yarn with a carbon matrix produced from a 15V pitch precursor. Those changes were monitored by microscopy and elastic properties measurements using vibrating beam techniques.

II. EXPERIMENTAL

Creep experiments were conducted with a specially modified high temperature furnace.[1] Samples of unidirectional carbon-carbon composite yarn were prepared from HM3000 yarn and 15V pitch by immersing yarn in melted pitch, then calcining it up to 1000°C at a slow heating rate under inert atmosphere. The looped-end samples were then tested in tension in the creep apparatus. Samples exhibited creep rates increasing with temperature above 2000°C and failed below 3000°C. Scanning electron micrographs of a sample near the point of failure are presented in Fig. 1.

In contrast to other methods of measuring elastic properties using vibrational resonance,[2-4] we exploited the relatively good electrical conductivity of carbon fibers and of the unidirectional composite samples, both unpyrolyzed and fully heat-treated. Vibrational modes were excited by passing an oscillating current through the sample in the presence of a magnetic field of approximately 3 kG provided by a small cobalt-samarium alloy magnet. Resonance was observed by means of a low-power optical microscope. The experiment is diagrammed in Fig. 2. The beam is supported with pointed electrical contacts, reducing the number of physical contacts with the



HM3000-65 WT% 15V PITCH

$T_{MAX} \rightarrow 3000°C$

$\frac{\Delta L}{L}$ (hot zone) → 5%

10 ksi

Figure 1. Example of matrix cracking and filament necking.

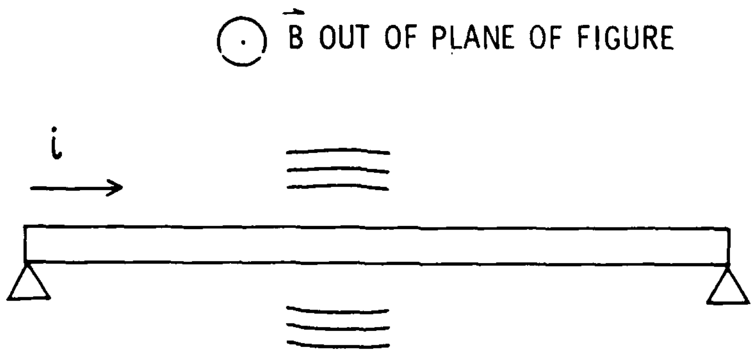


Figure. 2. Schematic of beam in transverse vibration.

sample to two from the four required for the piezoelectric technique. Simple vibrating beam theory was used for analyzing the results,[5] and beams of rectangular, round, and other cross sections were measured.

III. RESULTS AND DISCUSSION

After straining to failure at approximately 3000°C, the HM3000-15V unidirectional composite sample contained numerous transverse matrix cracks spaced regularly along the filament direction, similar to those in Fig. 1. Filaments within the matrix cracks narrowed substantially, by a factor of 3 or more, in contrast to the WCA-A240 and P55-A240 composite samples.[1] There is also a wide range in the degree of narrowing of filaments within the cracks, though some filaments were apparently unaffected. This difference may indicate variation in the degree of interfacial bonding between filament and matrix, as well as possible breaks in filaments within the composite. Thus, it would be possible for stress transfer to be nonuniform among the filaments, such that the stresses on the most narrowed filaments would be greater than those on the unaffected ones.

Interfacial debonding in these materials is evidenced by their ability to undergo substantial bending at room temperature without fracture. In contrast to WCA-A240 and P55-A240 composites, which undergo brittle fracture in bending at room temperature after undergoing high temperature creep, the HM3000-15V sample of Fig. 1 could be bent to approximately a 1-in. radius, as shown in Fig. 3, as a consequence of the transverse matrix microcrack structure and the weakened or modified interfacial bonding. Ostensibly, during bending, the strain is accommodated by sliding of filaments within the sections of matrix holding the filaments together, so that stresses on the filaments and matrix do not cause fracture. The transverse microcracks in the matrix take on a wedge shape, moving toward each other at the inner fibers and moving away from each other at the outer fibers. There seems to be enough interfacial friction that the sample remains somewhat rigid and will hold its shape upon being flexed back and forth several times. The amount of bending that can be accommodated by this structure can be estimated by calculating the radius of curvature when the microcracks form wedges that are just touching at the inner fibers. This radius R is approximately ab/2c (assuming a, b >> c), where a is bundle diameter, b is average distance between microcracks, and c is average microcrack width. For a = 1 mm, b = 0.31 mm, and c = 0.00463 mm (see Fig. 3), the minimum bend radius is about 34 mm, close to that observed.

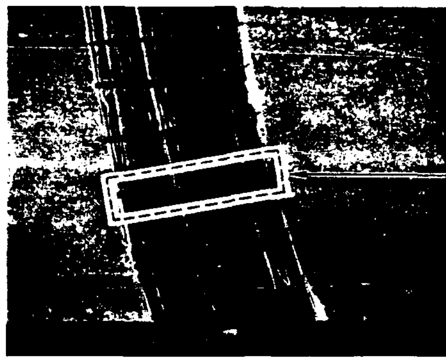


Figure 3. Postcreep sample of HM3000-15V showing bending and interfacial slipping at room temperature.

Elastic modulus along the fibers was calculated from the measured frequency and the dimensions and mass of the sample, using the formula for the lowest flexural mode of a simply supported beam:

$$f_1 = \frac{1.56\ (EI)^{1/2}}{(qL^4)^{1/2}} \qquad (1)$$

where EI is flexural stiffness (dyne-cm^2), q is mass per unit length (g/cm), and L is beam length (cm).[5]

Table 1 is a comparison of flexural modulus measurements for samples of P55-15V, both as-impregnated and after calcining at 1000°C under inert gas, and for HM3000-15V as-impregnated and after creep to 3000°C (see Fig. 1). Included are estimates of the modulus as a percentage of the rule-of-mixtures (ROM) modulus, neglecting the matrix modulus and assuming the following properties of the fibers: P55--10 μm diam., 55 Mpsi; HM3000--7 μm diam., 50 Mpsi. In contrast to the other samples, which have a substantial fraction of the ROM modulus, the apparent modulus of HM3000-15V has greatly decreased after high temperature creep, which may further indicate interfacial debonding, discussed above on the basis of the bending observations.

Table 1. Average Flexural Modulus

Specimen	Modulus (Mpsi)	%ROM	Diameter (mm)	Density (g/cm^3)
P55-15V				
As-impregnated	10.8	56	0.767	1.52
1000°C	17.1	76	0.701	1.23
HM3000-15V				
As-impregnated	11.2	35	0.48	1.52
3000°C	1.3	11	0.783	1.38

Greszczuk analyzed a partially debonded unidirectional composite and reported how the flexural stiffness may be lower than for a fully bonded composite.[6] Using this argument, and from a few simple geometric assumptions and by scaling Eq. (1), it can be established that a unidirectional composite, if debonded longitudinally into n identical independent yarn bundles, each of frequency f_1 and modulus E, has approximately the same frequency as a fully bonded composite with modulus E/n. By this argument, for example, the final sample in Table 1 would exhibit the equivalent frequency if it were fully bonded and had a modulus of 1.3 Mpsi or if it were debonded into n = 1/0.11 ~9 sub-bundles, each with ROM modulus of 11.8 Mpsi.

IV. CONCLUSIONS

High temperature creep of unidirectional carbon-carbon composites has been shown to give rise to changes in microstructure and apparent elastic modulus, which can indicate cracking and filament-matrix debonding.

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